Microstructure and Mechanical Property Study of Cu-graphite Metal Matrix Composite Prepared by Powder Metallurgy Route

A thesis submitted in partial fulfillment
of the requirements for the degree of

Master of Technology

in

Metallurgical and Materials Engineering

by

Chandana Priyadarshini Samal

Department of Metallurgical and Materials Engineering
National Institute of Technology Rourkela
Rourkela, Orissa, 769 008, India

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Chandana Priyadarshini Samal
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under the guidance of

Prof.Debasis Chaira **Prof.Bipin Bihari Verma**

Department of Metallurgical and Materials Engineering
National Institute of Technology Rourkela
Rourkela-769 008, Orissa, India

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NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

CERTIFICATE

This is to certify that the thesis entitled **"Microstructure and Mechanical Property Study Of Cu-graphite Metal Matrix Composite Prepared by Powder Metallurgy Route"** submitted by **Chandana Priyadarshini Samal (210MM1252)** in partial fulfilment of the requirements for the award of **MASTER OF TECHNOLOGY** Degree in **Metallurgical and Materials Engineering** at the **National Institute of Technology, Rourkela** (Deemed University) is an authentic work carried out by her under our supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

Date: 25th May, 2012

Dr. Debasis Chaira
Dept. of Metallurgical & Materials Engg.
National Institute of Technology
Rourkela -769008

Prof. B. B. Verma
Dept. of Metallurgical & Materials Engg.
National Institution of Technology
Rourkela-769008

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"The will of God will never take you where Grace of God will not protect you."

Thank you God for showing me the path. . .

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Chandana Priyadarshini Samal

ABSTRACT

Copper–graphite metal matrix composites possess the properties of copper, i.e. excellent thermal and electrical conductivities, and properties of graphite, i.e. solid lubricating and small thermal expansion coefficient. Copper matrix containing graphite are widely used as brushes, and bearing materials in many applications due to the excellent thermal and electrical conductivities, and the favorable self-lubricating performance. The addition of solid lubricant particles into a metal matrix improves not only the anti-friction properties, but also wear and friction properties. In the present investigation, attempts have been made for the fabrication of Cu-graphite MMC by conventional and spark plasma sintering (SPS) techniques.

Copper-graphite MMCs were fabricated by mixing 1, 3, 5, and 10 vol. % of graphite powder into copper powder followed by conventional powder metallurgy route. The composite powder mixture were cold compacted by uni-axial press and then sintered in tubular furnace using argon gas. In another set of experiments, Cu-1 vol. % graphite and Cu- 5 vol. % MMCs were fabricated by spark plasma sintering technique at 700°C under vacuum for 5 minutes. The MMCs were characterised by x-ray diffraction (XRD) and scanning electron microscopy (SEM). Different mechanical properties like density, bulk hardness and wear study were also conducted. XRD spectra show the presence of Cu, graphite and Cu_2O peaks which shows that no interaction between Cu and graphite takes place during fabrication. The presence of a weak peak of Cu_2O proves that slight oxidation of Cu takes place during conventional sintering of MMCs. However, no peak of Cu_2O is visible for SPS as it was conducted under vacuum. It has been found that addition of graphite into copper does not result in much improvement in hardness due to the soft nature of graphite. However, 90 and 97 % of theoretical density have been obtained for conventional sintered and SPS samples respectively. Maximum Vickers hardness value of around 100 has been achieved for Cu-1 vol. % graphite MMC when it is fabricated by SPS. However, a hardness value of 65 has been obtained for the same composite when it is fabricated by conventional sintering at 900°C for 1 hour. To study the effect of milling, Cu-1vol. % graphite and Cu-5 vol. % graphite powder mixture were milled for various time periods and then sintered. It has been found that hardness increases with milling. The micrographs of Cu-graphite reflect the clean interface and good compatibility between matrix and reinforcement. It has also been found that graphite particles are uniformly distributed into copper matrix. From wear study, it is

concluded that the wear resistance of the composite increases with increase in graphite content due to the lubricating properties of graphite. It has also been found that wear depth decreases with increase in graphite content. SPS sintered samples show higher wear resistance than conventional sintered samples. It has also been found that compressive strength increases with addition of graphite and maximum up to 3 vol. % of graphite. With further addition of graphite there is a decrease in compressive strength due to increase in brittle nature of composites.

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Introduction

CHAPTER 1

1.1 Introduction

The particulates reinforced metal matrix composite (MMC) is one of the new structural materials, and a rapid development can be seen in recent years because of excellent properties and wide application prospects in the near future. For several years research on fabrication methods and material property estimations for particulates reinforced metal matrix composites has been one of the focuses in composite fields, and many excellent research results have been obtained. Various materials have been combined with each other and give intended properties and are different from their base materials. Such composite materials make this concept true and reinforcement in a matrix of this material contributes enhancement properties. But, neither matrix nor reinforcement alone but only MMC can able to fulfil the requirement. MMCs are exciting materials which find increasing applications in aerospace, defence, transportation, communication, power, electronics, recreation, sporting, and numerous other commercial and consumer products. Rapid advancement in the science of the fibres, matrix materials, processing interface structure, bonding and their characteristics on the final properties of the composite have taken place in the recent years. Even though they have recently used but have more tremendous effect due to their useful properties like specific strength, specific stiffness, wear resistance, corrosion resistance and elastic modulus etc.

Copper is mostly used industrial and functional metal for thermal and electronic packaging, electrical contacts and resistance welding electrodes as it has very good electrical and thermal conductivity. During the operation of a large class of electrical machinery one is faced with the problem of transferring electric current from a stationary conductor to another conductor moving relative to it. The chief method of current transfer used today is that in which one conductor slides on the other, the current being transferred across the sliding interface. This form of contact at once imposes a conflict of requirements; on the one hand a large contact force is desirable to maintain effective current transfer, whilst on other hand it is advisable to have as small a contact force as possible in order to reduce the wear of the sliding components.

Two of the main problems associated with the operation of sliding contacts are the transfer of current across the sliding interface and the wear of the material. These two problems cannot be treated in isolation from one another; not only is the rate of wear clearly dependent upon the current passing but the manner in which the contacts wear can, in certain circumstances, influence the mode of current transfer.

Copper–graphite particulate composites possess the properties of copper, i.e. excellent thermal and electrical conductivities, and properties of graphite, i.e. solid lubricating and small thermal expansion coefficient. Copper matrix containing graphite are widely used as brushes, and bearing materials in many applications due to the excellent thermal and electrical conductivities, and the favorable self-lubricating performance.[1] Copper-graphite with low percentages of graphite is also used for slip rings, switches, relays, connectors, plugs and low voltage d.c. machines with very high current densities. For lower current densities and better cooling conditions, higher percentages of graphite are also used because of their lower wear rate. It has been reported that the addition of solid lubricant particles into a metal matrix improves not only the anti-friction properties, but also wear and friction properties.

Most of the bearing alloys that are presently used contain a soft phase like lead, which give the required anti-friction property. Due to its harmful effects, restrictions have been imposed on the use of lead. This has prompted researchers to find alternative materials, which impart tribological properties similar to those of lead. Certain metal matrix composites (MMCs) containing soft particles have been investigated for tribological properties. These MMCs have not only reduced friction but also lead to reduced wear of the counterface. Faced with all these challenges like low mechanical properties of pure Cu, Cu based alloys and harmful effects of Pb, a suitable alternative material copper-graphite metal matrix composite has been developed in the present investigation.

Although certain metals, metal oxides and non-metallic materials have desirable characteristics for contact applications, such as erosion and welding resistance, but are of low conductivity. Combination of these with copper, to give acceptable conductivity, should therefore produce a material with optimum properties. Unfortunately the metals concerned are of high melting point and do not alloy with copper, consequently it is not possible to produce them by conventional melting techniques. This also applies to the metal oxide and non-metal combinations with copper. The only viable manufacturing procedure at room temperature to produce such combinations is powder metallurgy.

1.2 Aims and objectives of the present study

- Fabrication of copper-graphite MMC by powder metallurgy route using Cu and graphite as starting materials
- Improvement of mechanical properties of the composite by using different sintering techniques such as conventional and spark plasma sintering
- Effect of various sintering parameters like temperature, time and pressure etc. and optimization
- Physical property like density measurement of sintered composite by Archimedes principle.
- Effect of milling graphite and copper powder to improve interfacial bonding
- Study the interface between Cu and graphite
- Failure analysis of the fabricated composites by compression and 3-point bend tests
- Improvement of wear resistance of the fabricated composites

1.3 Scope of the thesis

This organization of the thesis is as follows. The first chapter presents the importance and objectives of the present investigation. The second chapter gives a brief on metal matrix composites and different fabrication routes of MMC. It also presents a brief overview of literature review on fabrication of Cu-graphite MMC. A detailed experimental procedure and details of the various experimental techniques are provided in chapter 3. The fourth chapter presents about the results and discussions. A summary of the main findings along with conclusions is presented in chapter five. The next two chapters discuss about the scope of the future work and references respectively.

Chapter-2

Literature Review

CHAPTER 2

Literature Review

2.1 Introduction

Composite materials are structured material composed of two or more materials by (reinforcing element, filler and composite matrix binders) differing in the form of composition on a macro scale. The constituents retain their identities and they don't dissolve and merge completely into one another. The matrix combined with reinforcement improves different properties like physical, mechanical and wear properties etc. The combined material exhibits better strength than the individual one. The demands on material performance are so great and diverse. These materials offer the advantage of flexible design that can be tailored to the design requirement. In principle, composites can be fabricated out of any combination of two or more materials—metallic, organic or inorganic; but the constituent forms are more restricted. Composite materials are said to have two phases. The reinforcing phase is the fibres, sheets, or particles that are embedded in the matrix phase. The reinforcing material and the matrix material can be metal, ceramic or polymer and these materials are strong with low densities while the matrix is usually a ductile or tough material. The combination of matrix and reinforcement also depends upon strengthening mechanism. If the composite is designed and fabricated correctly, it combines the strength of the reinforcement with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material. Some composites also give the advantage of being tailorable, so that properties such as strength and stiffness can easily be changed by changing amount or orientation of the reinforcement material. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the reinforcement via the interface. Composites may be dispersion strengthened; fibre reinforced and particulate reinforced depending upon strengthening mechanism. For a composite to be isotropic in a specific property, such as CTE or Young's modulus, all reinforcing elements, whether fibers or particles have to be randomly oriented. This is not easily achieved for discontinuous fibers, since most processing methods tend to impart a certain orientation to the fibers. Composites differ by their matrix type, reinforcement type, size and form, composition, temper state, etc.

There are three broadly classified groups of composites: polymer matrix composite (PMC), metal matrix composite (MMC) and ceramic matrix composite (CMC).

2.2 Metal matrix composite

Metal Matrix Composite is an interesting engineering material due to its more demanding nature and their characteristics click the most all applications in engineering field. Metal-matrix composites (MMC) are of particular interest to materials scientist since they retain high strength and stiffness up to temperatures above 300°C and at the same time they have a low density. They are widely used for high-temperature industrial applications. Several studies have focused on dimensional changes and on chemical and interfacial stability between the constituents of unidirectional MMC resulting from thermal cycling. Such materials are well known for their exceptional high modulus, stiffness, wear resistance, fatigue life, strength-to-weight ratios, tailorable coefficient of thermal expansion etc. With these enhancements in properties, they pose for strong candidature for replacing conventional structural materials. Metal matrix composite has emerged as a class of materials capable of advanced structural, aerospace, automotive, electronic, thermal management and wear applications. The range of MMC applications is very large. Some of the important metal matrix composite components are applied and used as insulation materials for electrical construction, supports for circuit breakers and printed circuits, armors, boxes and covers, antennas, radomes, tops of television covers, cable tracks, wind mills, housing cells, chimneys, concrete molds, domes, windows, partitions, doors, and furniture. Automotive engineering parts like automotive body parts, wheels, shields, radiator grills, transmission shafts, suspension springs, chassis, suspension arms, casings, highway tankers, isothermal trucks, trailers, wagons, doors, seats, interior panels, and ventilation housings. In marine transports, it is used to fabricate hovercrafts, rescue crafts, patrol boats, trawlers, landing gears, anti-mine ships, racing boats and canoes. In air transports, MMCs are used as passenger aircrafts, composite gliders, leading edges, ailerons, vertical stabilizers, helicopter blades, propellers, transmission shafts and aircraft break discs. For space transports, it is used to make rocket boosters, reservoirs, nozzles and shields for atmosphere re-entrance. Some of the general mechanical applications are as gears, bearings, housing and casings, jack body, robot arms, fly wheels, weaving machine rods, pipes, components for drawing table, compressed gas bottles, tubes for offshore platforms, and pneumatics for radial frames. It is widely applied in sports and recreation industries to manufacture tennis and squash rackets, fishing poles, skis, poles used in jumping, sails, surfboards, roller skates, bows and arrows, javelins, protection helmets, bicycle frames, golf balls and golf sticks, and oars.

The advantage of metal matrix composites is their tailored mechanical, physical and thermal properties that include low density, high specific strength, high specific modulus, high thermal conductivity, good fatigue response, control of thermal expansion and high abrasion and wear resistance. Metal-matrix composites offer increased service temperature and improved specific mechanical properties over existing metal alloys. They consist of a ductile, usually low-density, matrix reinforced with elastic or ductile and strong fibers.

In a material composite, when the matrix is a metal or alloy, then it will give a metal matrix composite (MMC= Metal Matrix Composite).The performance of this materials i.e. their characteristics in terms of physical and mechanical peculiarity, depend on the nature of two components (chemical composition, crystalline structure and in the case of reinforcement, shape and size), the volume fraction of the adopted reinforcement and production technology. Generally the metal matrix composites utilize at the same time the properties of the matrix (light weight, good thermal conductivity, ductility) and of reinforcement, usually ceramic (high stiffness, high wear resistance, low coefficient of thermal expansion). By this way it is possible to characterize, if compared to the basic metal component, by high value of specific strength, stiffness and wear resistance, fatigue resistance and creep, corrosion resistance in certain destructive environments.

Metal matrix composite can be classified into various ways. One classification is the consideration of type and contribution of the reinforcement in particle, layer, fiber and penetration composite materials. Fiber composite materials can be further classified into continuous fiber composite materials (multi and mono filament) and short fibers or rather whisker composite materials. The reinforcement material of metal matrix composite is represented in the form of continuous fibers, short fibers. The structure and properties of the interface between a reinforcement and matrix play a major role in the mechanical and physical properties of metal matrix composites. Those composites with weak interfaces generally have relatively low strength and stiffness but high fracture toughness, while a strong interface usually leads to greater strength and stiffness but poorer fracture resistance. A medium strength bonding interface can make composites have high strength and stiffness as well as fracture toughness. These properties are related to the ease of de bonding and pull-out of fibers from the matrix during crack propagation. The properties of the interface in composites depend on the interface bonding between the fiber and matrix. This bonding is due to the nature and surface properties of the reinforcement and matrix **[3].** The discontinuously reinforced metal matrix composite is one of the new structural materials, and a rapid development can be seen in recent years because, this kind of composite has many excellent properties and wide application prospects in the near future. For several years research on fabrication methods and material property estimations for discontinuously reinforced metal matrix composites (especially aluminium metal matrix composites) has been one of the focuses in composite fields, and many excellent research results have been obtained. Along with the rapid development in research and the great potential needs in application, however, the consequent problems in discontinuously reinforced metal matrix composites both in academic and applied aspects concerned with its secondary processes (for example the machining and the joining) have been put forward. Being a new kind of structural material, as with any other engineering material, the widely successful application of this material is also directly dependent upon the research and development in the relevant fields, such as the joining and tooling technologies **[4].** In a recent review the processing of discontinuously-reinforced metal-matrix composites was discussed together with their mechanical properties. Systems where interactions between fiber and matrix occur either during fabrication or during high-temperature application are usually thermodynamically unstable. Good adhesion properties between fibre and matrix are achieved by the necessary wetting. Good wetting conditions are provided by strong bonding between host and fibre atoms. This interaction may result in undesirable effects: (1) dissolution of the fibre or particle in the matrix or (2) the formation of uncontrolled, undesired and brittle compounds **[2].** Chemical interaction and reaction between the matrix and reinforcement component determine the interface adhesion, modify the characteristics of composite component and affect the mechanical characteristic significantly. The formation of the interface between the matrix and reinforcing phase has a substantial influence on the production and characteristics of the metallic composite materials. The adhesion of both the phases is determined due to interaction between them. During production of molten matrix e.g. by infiltration, wetability becomes significant.

2.2.1 Improvement of different property in metal matrix composite

The tabulated form of different properties and advantages that developed in fabrication of this composite are given below in Table 1

Table 1 Different properties and advantages of metal matrix composite

	Better strength-to-density ratios
Property	Better stiffness-to-density ratios
	Good fatigue resistance
	Better elevated temperature properties o Higher strength o Lower creep rate
	Lower coefficients of thermal expansion
	Better wear resistance
Advantages	Higher temperature capability
	Fire resistance
	Higher transverse stiffness and strength
	No moisture absorption
	Higher electrical and thermal conductivities
	Better radiation resistance
	No out gassing
	Fabric ability of whisker and particulate-reinforced MMCs with conventional metalworking equipment.

2.2.2 Various strengthening mechanisms in MMCs

The various strengthening mechanism are given in schematically as follows:

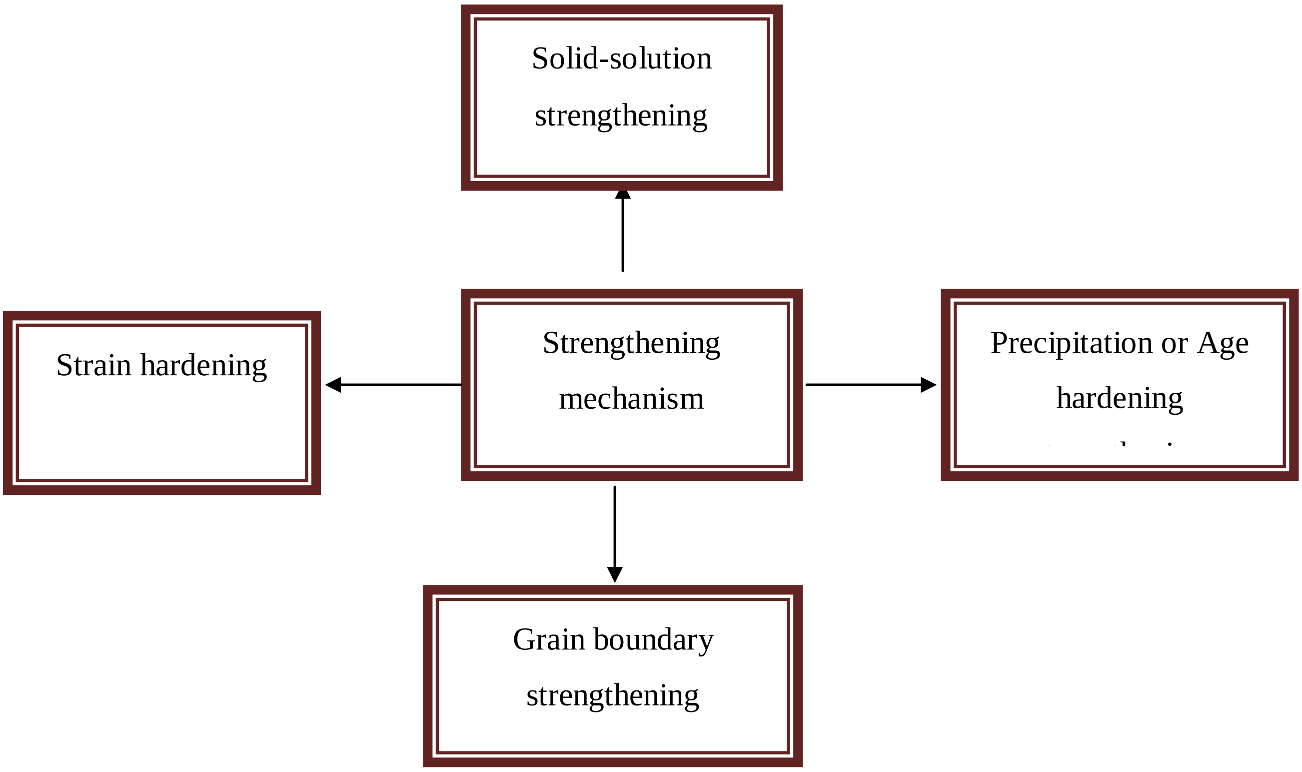


Table 2 Various strengthening mechanisms of MMC

Strengthening mechanism	Properties
Grain boundary strengthening	Two important ways by which grain boundary that acts as a obstacle to dislocation motion are (a) two different grain orientations (b) discontinuity of slip planes from one grain to another. Finer grain materials are harder and stronger due to more amount of grain boundary.
Solid-solution strengthening	In this case solute atoms give more impact on the frictional resistance to dislocation motion and with increase in solute atoms strengthening effect increases.
Strain hardening	Due to plastic deformation the material becomes harder and stronger. And dislocation density increases with deformation and cold working.

Precipitation or Age hardening	The small particles of new phase precipitate in matrix which harden material by creating obstacles to dislocation motion.

2.3 Fabrication of metal matrix composite

In general the most common manufacturing MMC technologies are divided primarily into two main parts: primary and the secondary. The primary processing is the composite production by combining ingredient materials (powdered metal and loose ceramic particles, or molten metal and fibre preforms), but not necessarily to final shape or final microstructure and the secondary processing is the step which obviously follows primary processing, and its aim is to alter the shape and microstructure of the material (shape casting, forging, extrusion, heat treatment, machining).[20-25] Secondary processing can change the constituents (phase, shape) of the composite. So, MMCs can be made by different ways like

- Solid state processing
- Liquid state processing
- Vapour state processing
- Plasma /spray deposition
- In situ processing

2.3.1Solid state processing

In solid state processing, the reinforcement is embedded in matrix through diffusion phenomena at high pressure and high temperature. It appears crucial monitoring of the diffusion phenomena to avoid the growth of undesirable phases or compounds species on interfaces. The various steps of processing are usually preceded by a "pre-processing" for the purpose of preparing the surface. The most common solid phases are based on powder metallurgy techniques. These typically involve discontinuous reinforcement, due to ease of mixing, blending and effectiveness of densification. The ceramic and metal powders are mixed isostatically, cold compacted and hot pressed to get full density. Then the fully dense compact typically undergoes a secondary operation such as extrusion, forging.[26-30]

Solid state processing method is followed by two ways:

1. Diffiusion bonding

2. Powder metallurgy

2.3.1.1Fabrication of MMC by diffusion bonding

This technique is mainly for the typical composite fiber consists of application of pressure and high temperature. One of these techniques is such that the "foil-fiber-foil" where alternating sheets of reinforcement (usually a long fiber) and matrix is greater than the other and then be united together. The consolidation of foil with fiber together, with the penetration of the metal, occurs through a process of sintering, which is implemented by two main phenomena. [30-35]

The first phenomenon is the creation of the matrix sheets deformation due to the mechanism of viscous plastic flow at high temperature, responsible for the penetration among the layers of fibers and winding. Another phenomenon is joining mechanism which occurs at the interfaces when layers come in contact. The fibers lined, next to one another can be consolidated by using different methodologies, in order to compose the layers of reinforcement.

Diffusion bonding is a really good method to produce composite with high mechanical properties. The main problem is the requirement of high intensity of energy (high pressures and high temperature).

2.3.1.2 Fabrication of MMC by powder metallurgy

The powder metallurgy is one of the popular solid state methods used in production of metal matrix composites. Powder processing involves cold pressing and sintering, or hot pressing to fabricate primarily particle- or whisker-reinforced MMCs. The matrix and the reinforcement powders are blended to produce a homogeneous distribution.[36-38] Powder metallurgy process is mainly consists of three phases. These are explained as below

(1) The first phase gives the preparation of the powder that is the constituents of the mixture and it follows the successive stages. One of the most popular methods for implementation of this phase is the "gas atomization" in which a gas liquid vein is directed by a gas jet at high pressure that broken it into small spherical drops. Thus the powder products have spherical morphology, good slider and packaging.

(2) In the second phase the powder products are mixed together with the reinforcement ceramics or other particles and then compacted in the desired forms (phase blending/milling). However, obtaining a uniform mixture during mixing is difficult, especially with the whiskers, which tend to entangled in clusters hardly refillable by the matrix particles. Agglomeration is formed due to the relative size of the particle. Despite the process effectiveness it is important to put some attention about the possibility of contamination of grinding, hammers and containers, as well as the presence of the reactive gas, that however also eliminated by the use of an atmosphere.

(3) The third process is the process of consolidation, during which the powders of worked mixture are welded together by sintering to form final product. During this process compression is conducted at a temperature as high as possible in order to bring the matrix in its most malleable state, through the movement the establishing the conditions of the movement of dislocations, but without causing the presence of liquid phase, which would adversely affect the mechanical properties of the product, cause to the segregation on grain and the formation of harmful inter metallic compounds. Nevertheless a small amount of liquid metal allows a pressure reduction required to complete consolidation i.e. without the porosity of the powder mixture. [39-41]

2.4 Fabrication of Cu-graphite MMC by powder metallurgy route

Several researchers have fabricated Cu-graphite MMC by powder metallurgy route. A brief of the literatures available is presented here.

S.F. Moustafa et al. suggested that the Cu matrix Ni coated reinforced composites have higher relative density and lower porosity content than the uncoated composites, due to the good adhesion between the reinforcements and the Cu-matrix. Yield and compression strengths of coated reinforcement powders containing composites are superior to those of uncoated ones **[5]. D. H. He et al.** explains that the layer transfer function of CGCMs (Carbon Composite Materials) can reduce wear and provide protection to contact wires and are self-lubricating materials which also exhibit a special electrical conduction mechanism **[6].**

S.F. Moustafa et al. pointed that both Cu-coated and uncoated graphite composites exhibit the same wear mechanisms, namely, oxidation induced delamination, high strained delamination, and sub-surface delamination **[7]. V.V. Rao et al.** showed that thermal contact conductance increases, as a function of contact pressure and it is a weak function of mean interface temperature in case of Al_2O_3/Al–AIN MMC **[8]. X.C. Ma et al.** proposed that the wear loss increased with increasing normal stress and electrical current. Adhesive wear, abrasive wear and electrical erosion wear are the dominant wear mechanisms during the electrical sliding wear processes **[9]. K. H. W. Seah et al.** reported that the increase in compressive strength is due to graphite particle acting as barriers to the dislocations in the microstructure and with increasing the graphite content within the ZA-27 matrix results in significant increases in the ductility, UTS, compressive strength and Young's modulus, but a decrease in the hardness **[10]. S.F. Moustafa et al.** gives the idea about the densification of compacts fabricated from coated powders is much faster with 2.5 times than those made from uncoated powders. The Cu matrix Ni-coated reinforced composites have higher relative density and lower porosity content than the uncoated composites, due to the good adhesion between the reinforcements and the Cu-matrix. Yield and breaking compression strengths of coated reinforcement powders containing composites are superior to those of uncoated ones **[11]. C.S. Ramesh et al.** suggested that micro hardness and tensile strength of hybrid composites are higher as compared to the matrix copper. Increased content of hard reinforcement in the hybrid composites leads to enhancement in micro hardness and strength of the hybrid composites, however, ductility decreases **[12]. K. Rajkumar et al.** noticed that copper–graphite composites were effectively sintered using microwave hybrid heating without any crack. The finer microstructure with relatively smaller and round pores, resulted due to microwave heating, enhances the performance of the composite **[13]. C.S. Ramesh et al.** reported that the Ni-P coated Si_3N_4 particles reinforced Al6061 composites exhibited lower coefficient of friction and better wear resistance when compared with unreinforced alloy at all the loads and sliding velocities studied. Formation of the oxide at the interface plays a significant role in reducing both coefficient friction and wear rate **[14]. K. Rajkumar et al.** gives that hardness of hybrid composites is higher than the unreinforced copper.

Increased content of harder reinforcement (TiC) in the hybrid composites leads to enhancement in hardness. Hardness of hybrid composites is decreasing with the increase in graphite content. Wear rate and coefficient of friction of hybrid composites and unreinforced copper increases with increase in normal load and Wear rates and coefficient of friction of hybrid composites are lower than those of unreinforced copper. Wear rate of hybrid composites is reduced with increasing % TiC and % graphite, due to the cooperative effect offered by both the reinforcements. Coefficient of friction of hybrid composites is decreased with increase in % graphite reinforcement **[15]. S.K. Ghosh et al.** suggested that the specific wear rate increases with the decrease of reinforcement size for a certain volume percentage of SiCp **[16]. A. Fathy et al.** observed that the increasing strain rate from 10^{-4} s^{-1} to 10^{-2} s^{-1} increased compressive strengths of all tested nanocomposites. The wear rates of the composites increased with increasing applied loads or sliding speed. The wear rate of the monolithic copper is more than that of the nanocomposites **[17].** **A. Yeoh et al.** gives that the expansion of the cylindrical specimens is observed in both the longitudinal and lateral dimensions with the greatest expansions measured for those composites in the 50 vol. % copper-50 vol. % graphite ranges. Spheroidization is due to result of non-wetting between copper and graphite. The maximum expansion is observed at Cu-50 vol. % and such a composite presents the highest number of interfaces between the constituents **[18].**

2.5 Applications of copper base composite materials

The various applications of copper base composites are tabulated in Table 2.[19]

Table 3 Various applications of copper base composite materials

Cu + W	Electrical contacts, resistance welding electrodes, electrodes for automatic welding
Cu+ TiC	Electrical contacts, resistance welding electrodes, electrodes for automatic welding
Cu + C	Electrical brushes, sliding contacts, electronic-laser-computer sub-assemblies
Cu + Co	Elements of electronic systems
Cu and other reinforcing agents	Sliding rings, comutators, composite materials of the firs wall particles of nuclear reactors

2.6 Summary

This chapter summarises about introduction of metal matrix composites and its applications. It also presents about fabrication of MMCs by powder metallurgy method.

Chapter-3

Experimental

3.1 Synthesis of copper-graphite meal matrix composite

Synthesis of copper graphite MMCS were performed by conventional powder metallurgy route like blending, compaction and finally sintering etc. Synthesis of the composite was done by two sintering methods (conventional and spark plasma sintering) to study the difference in microstructure and mechanical properties of the fabricated composites.

3.1.1 Conventional sintering

Blending was carried out in pestle and mortar to ensure uniform distribution of graphite into copper. Starting materials Cu & graphite powder having purity 99% & 95% were used. Cu powders were mixed with graphite to prepare composite powder mixture of 1, 3, 5 and 10 vol. % of graphite. The composite powder mixtures were then cold compacted by uni-axial press and then sintered in tubular furnace using argon gas. Here different sintering parameters like compaction pressure, sintering time and sintering temperature etc. were varied and tabulated in Table 3.

Table 4 Different sintering parameters for conventional sintering

Sintering Temperature	950, 900, 750 $^{\circ}$C
Compaction pressure	700, 600, 800 Mpa
Holding time	1, 1.5, 0.5 h
Atmosphere	Argon
Heating rate	5 $^{\circ}$C/minute
Relaxation time in compaction	2 minutes

3.1.2 Spark plasma sintering

In another set of experiment, Cu-1 vol. % graphite and Cu- 5 vol. % graphite powder mixture were sintered by spark plasma sintering (Dr. SINTER LAB SPS syntax) at a temperature of 700°C for 5 minutes under vacuum at a heating rate of 80°C/minute.

3.1.3 Milling

To study the effect of milling on fabrication of Cu-graphite MMC, Cu-1 & 5 vol. % of graphite powder were milled. Milling was conducted in Pulverisette-5 planetary ball mill. The different milling parameters used during milling are represented in Table 4

Table 5 Various milling parameters used for milling

Mill type	Pulverisette-5 planetary ball mill
Milling time	0.5, 1 and 2 h
Wet milling	Toluene
Milling speed	300 rpm
Grinding media:	
Type	Ceramic (Zirconia)
Ball size (diameter)	15 mm
Ball to power ratio by weight	5:1
Jar volume	250 ml (Zirconia)

3.2 Microstructural characterization techniques used

3.2.1 X-ray diffraction (XRD) analysis

X ray diffraction of the composites was performed by using the X-ray diffractometer (PANalytical). X-ray diffraction patterns were recorded from 20° to 80° with a Philips X-pert MPD software using Cu K_α (λ=1.542A°) target with an accelerating voltage of 40 KV. Data were collected with a scanning rate of 2^0/minutes. The K_α doublets are well resolved.

3.2.2 Scanning electron microscopy (SEM) study

Morphology, particle size and micro structural characterization of sintered pellets were observed under a JEOL JSM-6480 LV scanning electron microscope. Both secondary electron (SE) and back scattered electron (BSE) image modes were taken on the basis of the requirement. Elemental analysis was done by EDX (Energy Dispersive X- ray) detector of Oxford data reference system. The best possible resolution microstructures were taken at suitable accelerating voltage.

3.2.3 Particle size analysis

Particle size of as received copper and graphite powder was measured by Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron). Powders were dispersed into a solution of 500 ml distilled water and 25 ml of sodium hexa metaphosphate.

3.3 Mechanical property study

3.3.1 Hardness measurement

Vickers hardness values of all the sintered specimens were calculated by using micro hardness tester LM248AT under 0.3kgf load and 5 second dwell time. For each specimen at least 5 measurements were taken at equivalent positions.

3.3.2 Density measurement

Density of the composites was measured by using Archimedes principle. First, the dry weight of the pellets was measured and then those were boiled in a hot plate to remove the pores. When the bubble creation was stopped, then the suspended weight and soaked weight of the pellets were calculated. Finally, densification parameter and % of theoretical density were calculated by using formula.

3.3.3 Transverse rupture strength (TRS) study

Transverse rupture strength samples were prepared by making standard transverse rupture strength test blocks (6.35x12.7x31.7 mm) according to ASTM B 312. After sintering for one hour at 900°C, TRS test was carried out on Instron-1195. Failure analysis of composites was done by observing fracture surface on SEM.

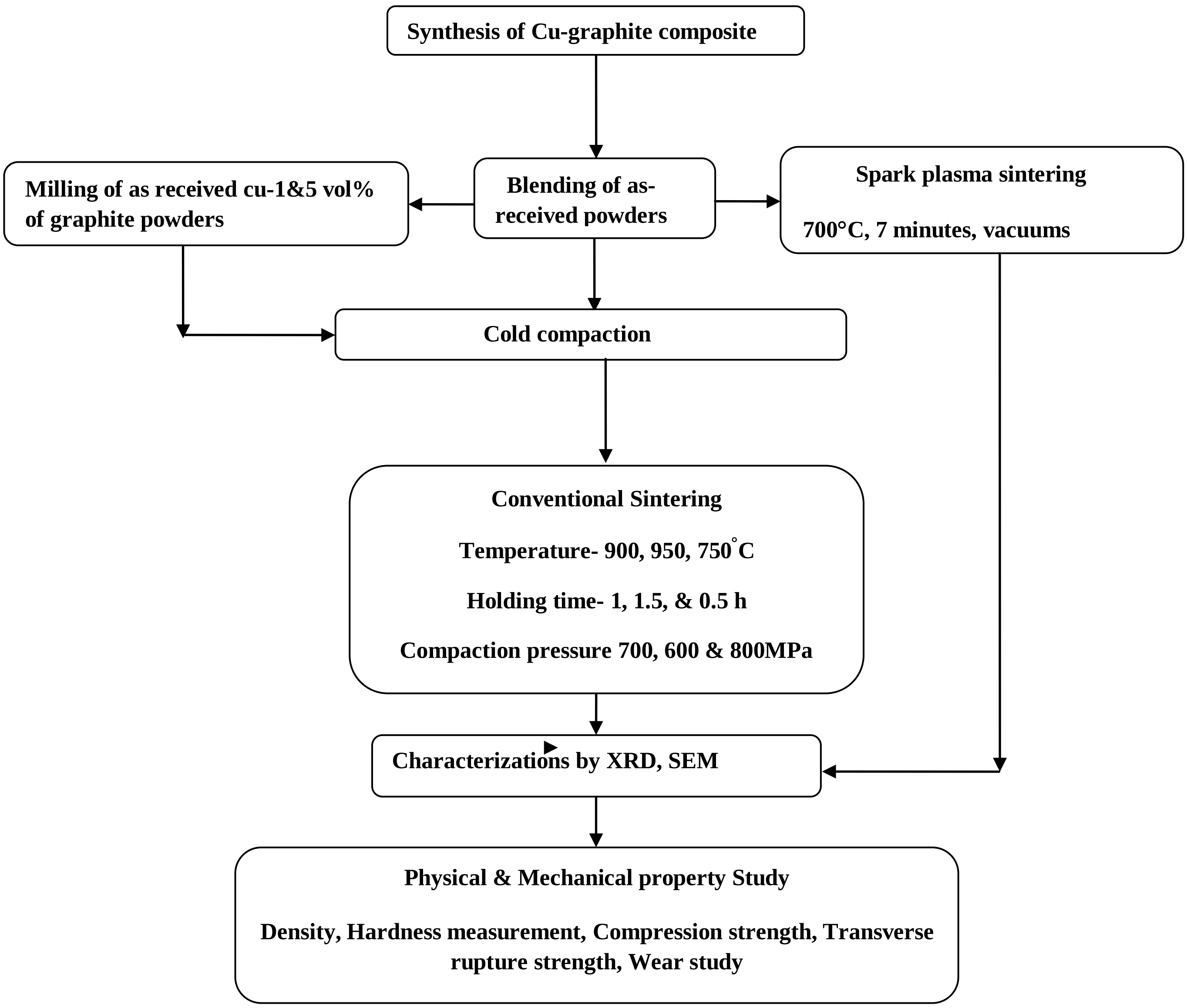
3.3.4 Compression test study

Samples for compression test were prepared by cold compaction and sintering maintaining L/D ratio > 0.8. Compression test was carried out on SATEC, 600KN INSTRON. The maximum compressive strength, % elongation and yield strength were measured.

3.3.5 Wear study

In present study, Ball-On-Plate Wear Tester (TR-208 M1) instrument was used to determine the wear behaviour of the conventional and SPS samples. This experiment was conducted at constant load of 20N for 15 minutes at 20 rpm. Stainless steel ball of 4 mm diameter was used for wear study.

The schematic representation of the experimental work is shown in flow diagram as follows:


Synthesis of Cu-graphite composite
Milling of as received cu-1&5 vol% of graphite powders
Blending of as-received powders
Spark plasma sintering
700°C, 7 minutes, vacuums
Cold compaction
Conventional Sintering
Temperature- 900, 950, 750˚C
Holding time- 1, 1.5, & 0.5 h
Compaction pressure 700, 600 & 800MPa
Characterizations by XRD, SEM
Physical & Mechanical property Study
Density, Hardness measurement, Compression strength, Transverse rupture strength, Wear study

Chapter-4

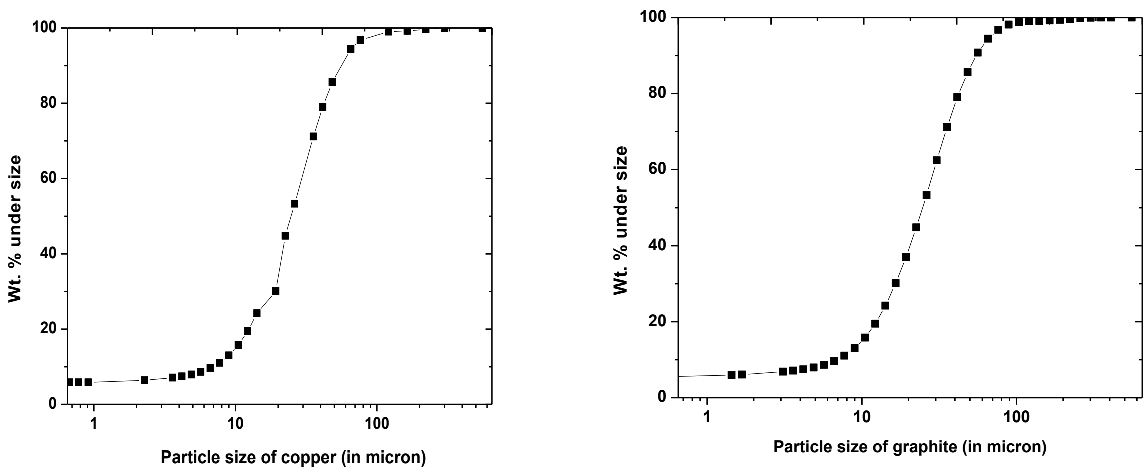
Results and Discussions

4.Result and Discussion

4.1. Characterization of as received powder

4.1.1Particle size analysis

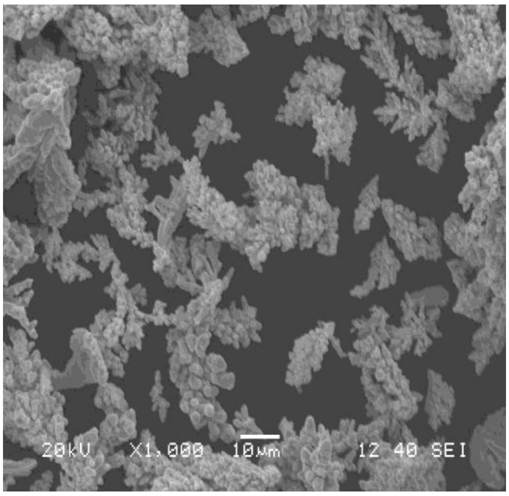
Particle size of the as received copper and graphite powders were examined by particle size analyzer. Figure 1(a) & (b) shows the particle size distribution. It can be seen that the average size for Cu is 24.73 microns and for graphite is 34.14 micron.



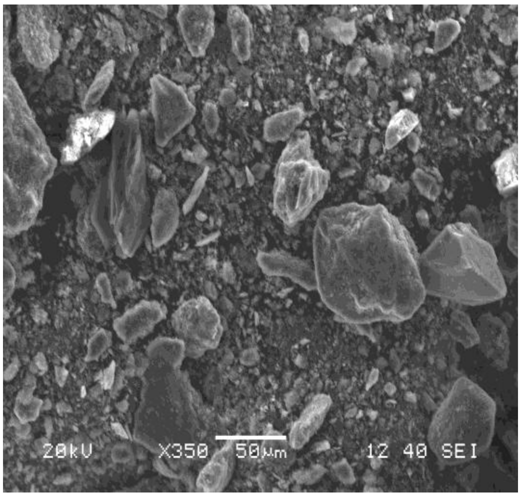
(a) (b)

Fig. 1 Particle size analysis of (a) copper & (b) graphite powders

Figure 2 (b) & (c) shows the SEM micrographs of as received Cu and graphite powders. The dendritic structure of copper & flaky shape of graphite powder can be seen from the micrographs.



(a)



(b)

Fig. 2 SEM Micrographs of (a) copper & (b) graphite powder

4.1.2 Characterization of Cu-graphite MMC

4.1.2.1 X-ray diffraction analysis

XRD analysis of copper-graphite MMC with different volume percentage of graphite was well conducted. The XRD spectra of conventional sintered at different temperatures and spark plasma sintered composites are shown in Fig. 3(a), (b), (c) & (d).

The XRD spectrum shows the presence of strong peaks of Cu and very weak peaks of Cu_2O and graphite in case of conventional sintering. A very weak peak of graphite is seen due to the occurrence of less amount of graphite. Some amount of copper oxide is developed due to the involvement of atmospheric oxygen during conventional sintering in the tubular furnace. It can be concluded from XRD spectra that during fabrication of composites, no reaction between copper and graphite takes place.

The XRD spectra of Cu-1 vol. % and Cu-5 vol. % graphite MMC is fabricated by spark plasma sintering at 700°C. Here, unlike conventional sintering no such formation of Cu_2O takes place. This is due to the fact that spark plasma sintering was carried out in vacuum. So, oxide formation is inhibited.

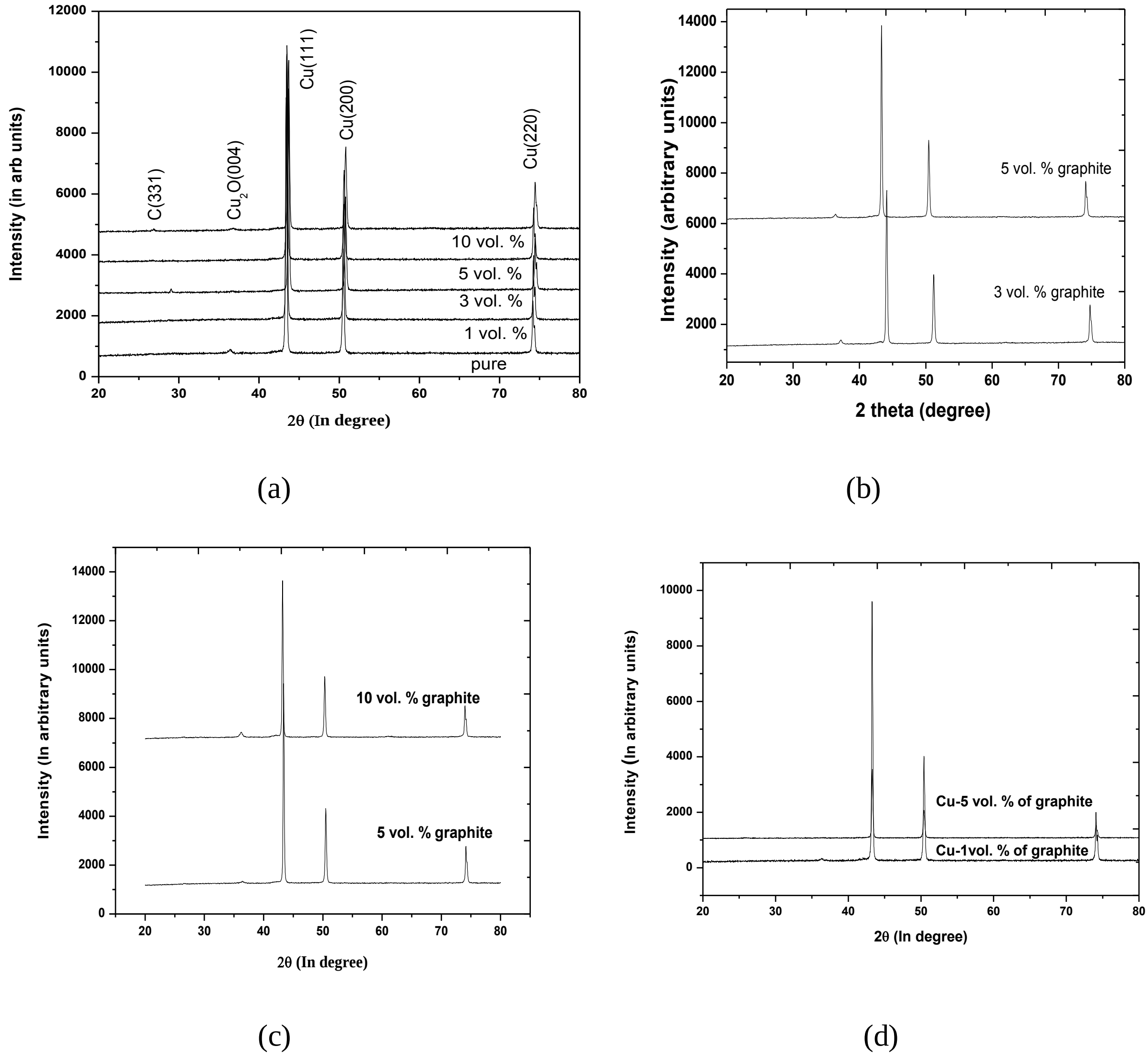


Fig. 3 XRD spectrum of cu-graphite MMCs conventionally sintered at (a) 900 (b) 950 (c) 750°C for 1 h and (d) spark plasma sintered at 700°C

4.1.2.2 Scanning electron microscopy

Fig. 4 (a) shows the representative FESEM micrograph of Cu-10 vol. % graphite composite conventionally sintered at 900°C for 1 h. It can be seen from the micrograph that graphite particles are uniformly distributed into Cu matrix. Fig. 4 (b-e) shows the FESEM micrograph, EDS spectra and distribution of Cu and graphite throughout the micrograph. The EDS spectra show the peaks of Cu and graphite. The corresponding distribution of Cu and graphite also can be seen.

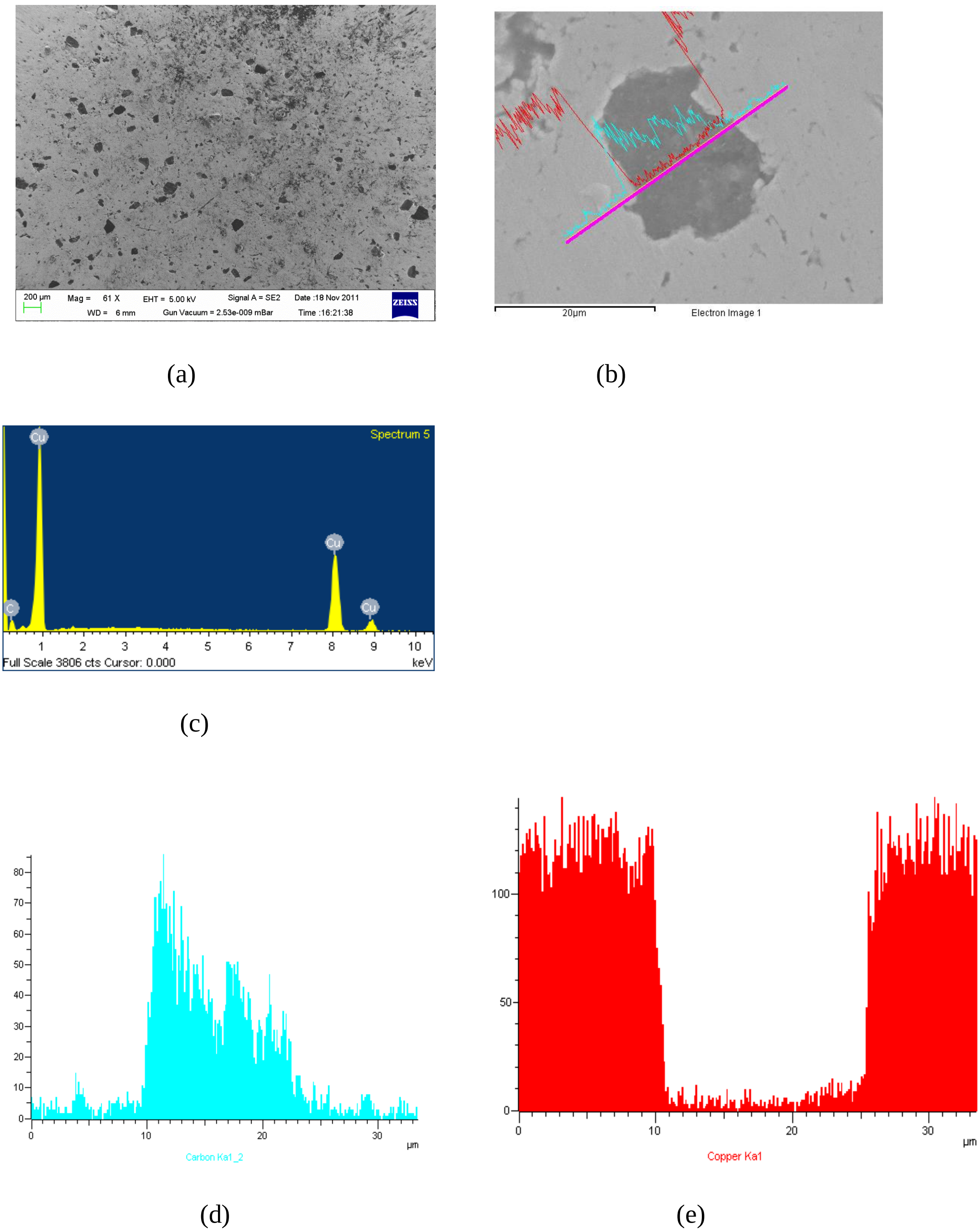
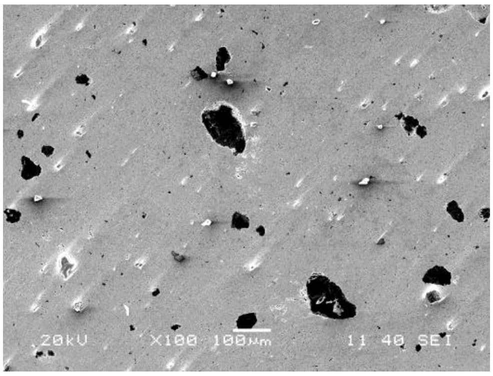
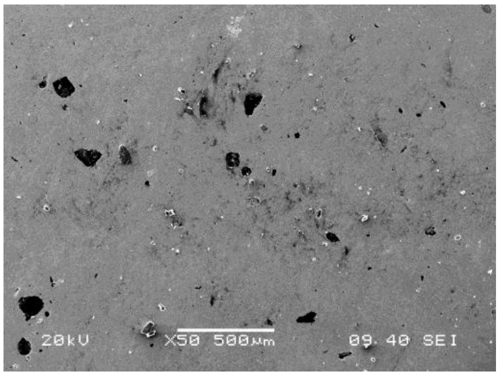


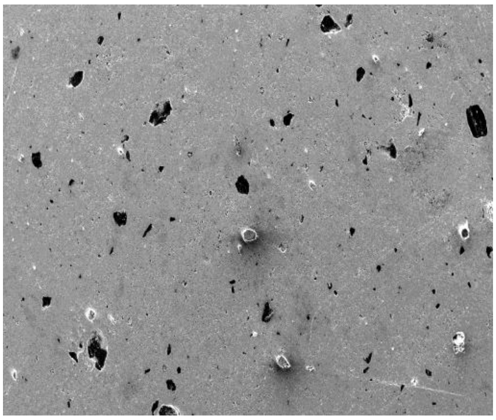
Fig. 4 FESEM micrographs of Cu-10 vol. % graphite MMC sintered at 900° C for 1 h

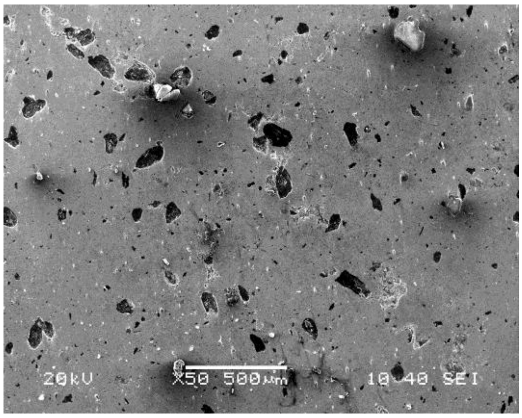
Figure 5 shows the SEM images of Cu with 1, 3, 5 & 10 vol. % of graphite reinforced MMC conventional sintered at 900°C for 1 h. From micrographs it is noticed the uniform distribution of graphite reinforcement (dark portion) into copper matrix (white portions). Also with increase in graphite volume percentage, number counts of graphite particles (black portion) increases which is evident from the micrographs.





(a) (b)

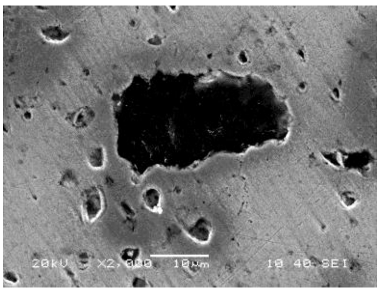


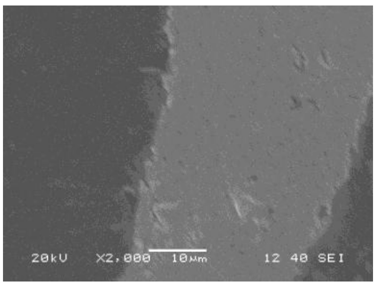


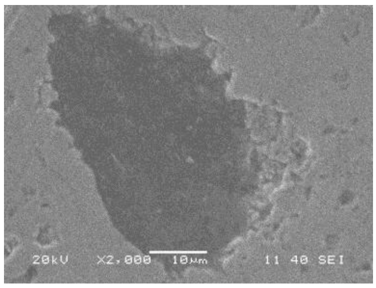
(c) (d)

Fig. 5 SEM micrographs of 1, 3, 5 and 10 vol. % graphite reinforced MMC conventionally sintered at 900°C for 1 h

Fig. 6 shows the SEM micrographs of the interface between Cu and graphite. It can be seen from the micrographs that no interfacial product is formed at the interface, confirms that no reaction takes place between matrix and reinforcements during fabrication.



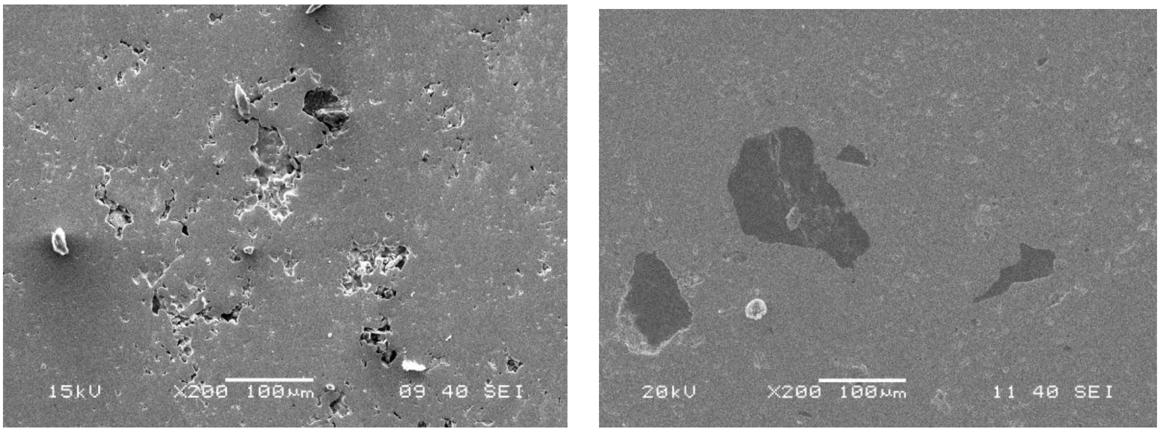




(a) (b) (c)

Fig. 6 Micrographs of conventionally sintered Cu-graphite MMC at (a) 900 (b) 750 (c) 950° C for 1 h

Fig. 7 shows the SEM micrographs of Cu- 5 vol. % graphite powder mixture cold compacted at 600 and 800 MPa and then sintered at 900°C for 1 h. It can be seen from the micrographs large amounts of pores and voids are present in case of 600 MPa as compared to 800 MPa. During cold compaction rearrangement and deformation of powder particles take place.

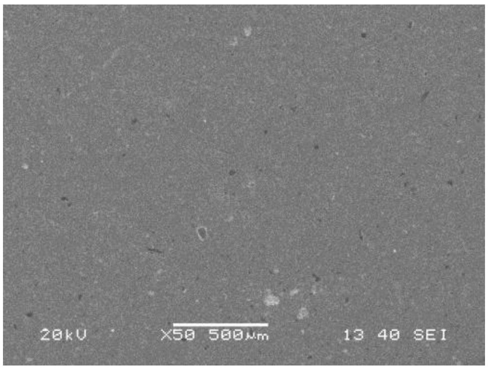


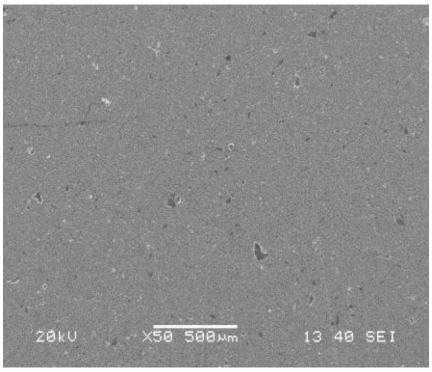
(a) (b)

Fig. 7 Cu-5 vol. % graphite composite cold compacted at (a) 600 and (b) 800 MPa and then sintered at 900°C for 1 h

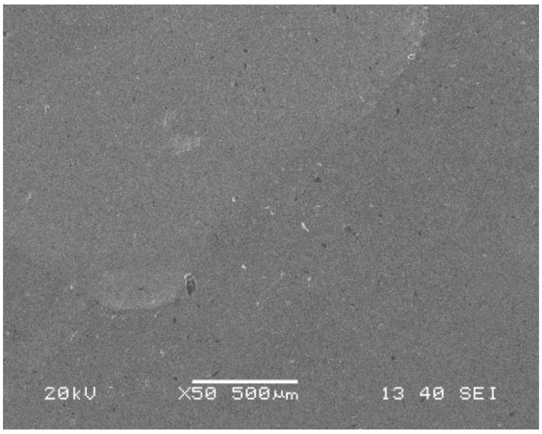
From micro structural point of view, proper interfacial bonding is found between matrix and reinforcement at all the three temperatures. At higher temperature the reinforcement is more induced into the copper matrix due to the soft nature of graphite. But in other temperature it is not so prominent. It shows better compatibility due to negligible amount of pores.

To produce a fine and homogeneous distribution of graphite particles with a very fine particle distribution into Cu, milling was conducted. It is also aimed to study the effect of metal-coated graphite (MCG) on the mechanical properties (strength and hardness) of consolidated and sintered composite materials. Fig. 8 shows the SEM micrographs of Cu-1 vol. % and Cu-5 vol. % graphite reinforced composite powder mixture milled for different time periods and then sintered at 900° C for 30 minutes. It can be seen from micrographs that graphite particles become fine as milling progresses and are homogeneously distributed.





(a) (b)



(c)

Fig. 8 SEM Images of Cu-graphite composite after milling for (a) 0.5h (b) 1h (c) 2h and then sintered at 900°C for 30 minutes.

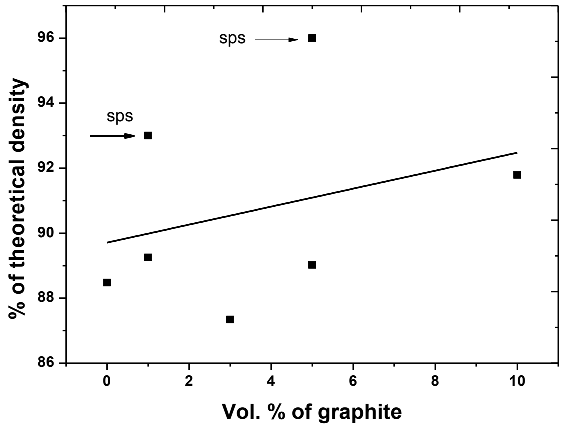
4.2 Physical properties

4.2.1 Density calculation

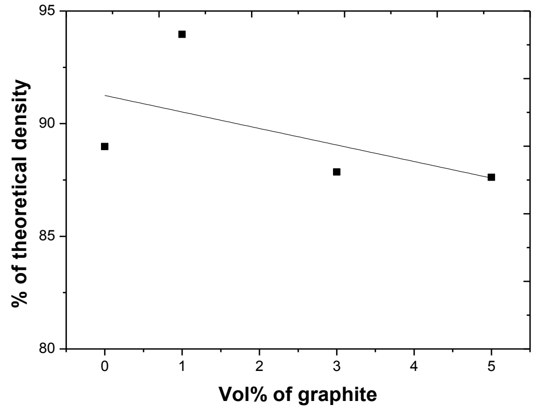
4.2.1.1 Effect of temperature

The variation of % theoretical density with increase in graphite volume percentage at different temperature is shown in figures Fig. 9 (a), (b) & (c). Higher density values are obtained for SPS samples as compared to conventional sintered at 900°C. The reason is due to the simultaneous applications of pressure and heat during SPS which results in higher hardness than conventional sintering. It is seen that density data are scattered and uneven. However, it has been found a density value of around 90% for conventional sintering and

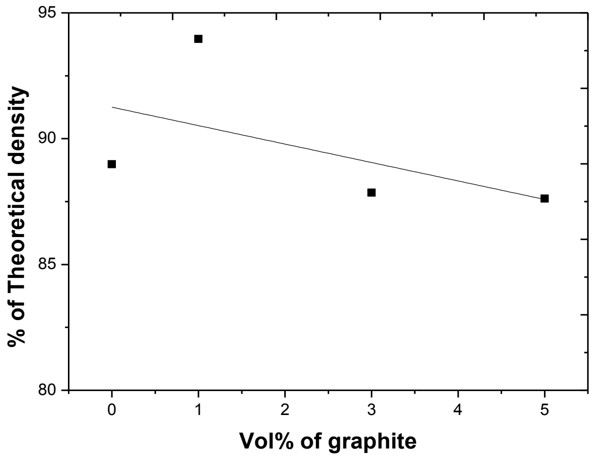
96% for spark plasma sintering. It is also observed that there is very little variation in density with temperature.



(a)



(b)

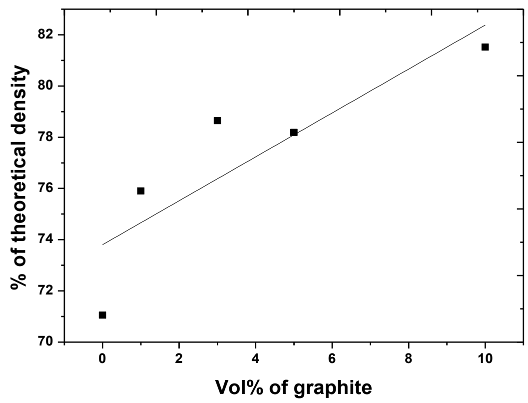


(c)

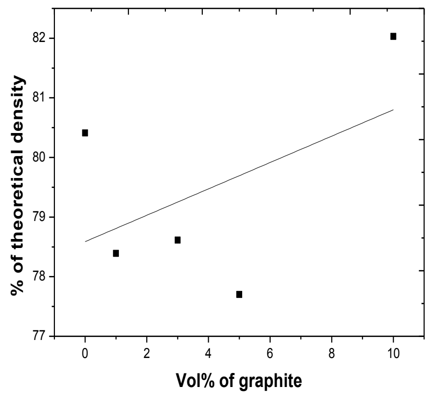
Fig. 9 Variation in density with graphite reinforcements at different temperature (a) 900 (b) 950 (c) 750°C

4.2.1.2 Effect of pressure

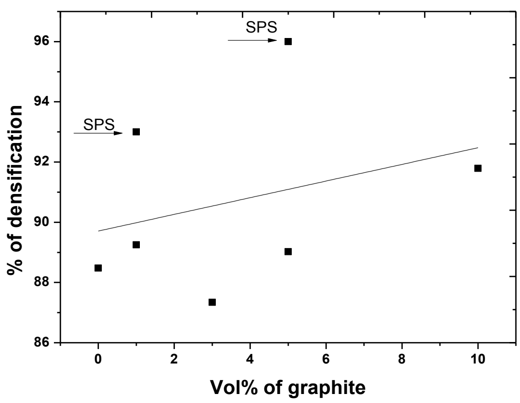
From the Fig. 10 (a), (b), & (c) shown below it is pointed that the maximum amount of density are seen in case of 700 MPa compaction pressure. So, it is the optimum compaction pressure. It also can be observed that SPS yield higher density than conventional sintering due to simultaneous applications of temperature and pressure. The maximum density of 96% is achieved in case of SPS as compared to 89% in case of conventional for 5 vol. % composite at a pressure of 700 MPa. However, the density data are scattered and uneven for different compaction pressure and hence no correlation can be established. The general trend of densification is that density value increases with graphite particle content due to soft nature of graphite. It should be noted that hard, brittle ceramic reinforcements prevents themselves and also shields Cu particles from plastic deformation when pressure is applied. Whereas graphite particles help to plastically deform ductile Cu and graphite are deformed and helps in increase of density value.



(a)



(b)

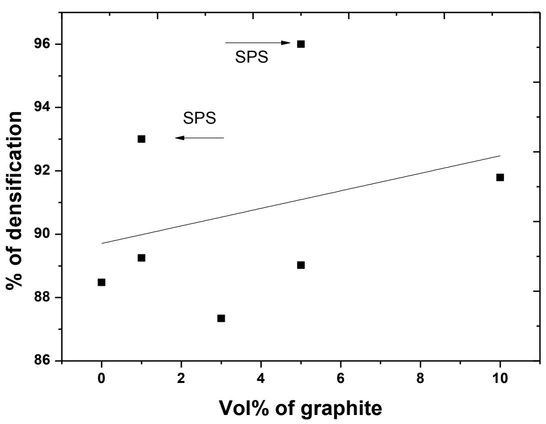


(c)

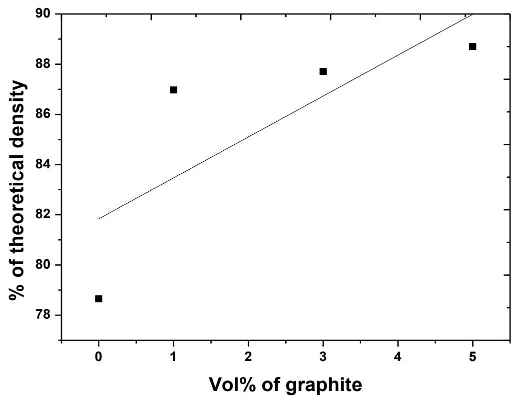
Fig. 10 Variation in density with graphite percentage at a pressure of (a) 800 (b) 600 (c) 700 MPa

4.2.1.3 Effect of time

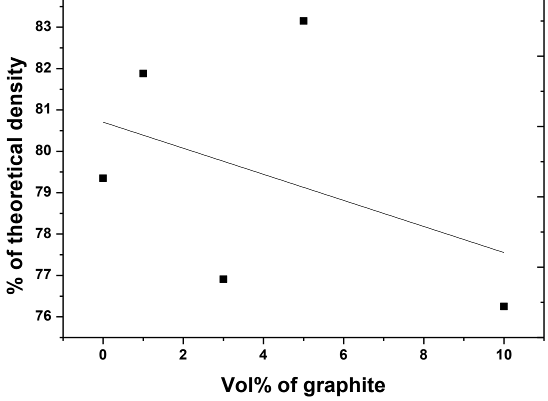
Fig. 11 shows the variation of density with time for different amount of graphite reinforcements. It is observed from the graph that higher density values are obtained for SPS as compared to conventional. The reason is due to the simultaneous applications of temperature and pressure during fabrication of MMC by SPS. It is also seen that density data are scattered and hence no correlation can be established with sintering time. It is also observed that a maximum density value of around 91% can be achieved for conventional whereas 96% can be achieved for SPS.



(a)



(b)



(c)

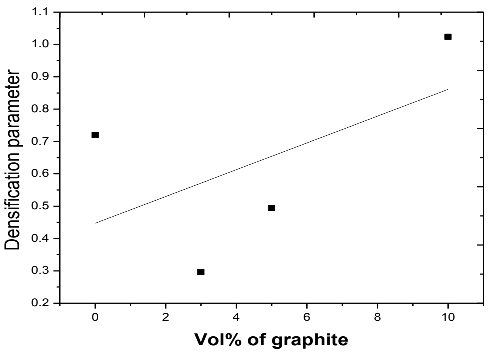
Fig.11 Variation in density with time at different percentage of graphite percentage: (a) 1 (b) 0.5 (c) 1.5 h

4.2.2 Densification parameter

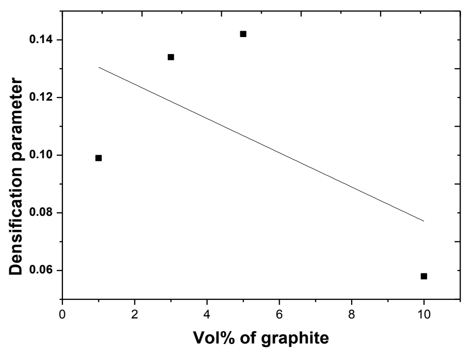
To get an idea about densification behaviours of Cu-graphite composites, densification parameter (DP) of the fabricated composites were calculated. We know,

$$\text{Densification Parameter (DP)} = \frac{\text{Sintered Density} - \text{Green Density}}{\text{Theoretical Density} - \text{Green Density}}$$

The variation of DP with graphite percentage at different temperatures is shown in fig. 12 From below three graphs we observe that densification parameter gives positive value and it increases with increase in graphite volume percentage. The positive DP values show the shrinkage of the composites after sintering.



(a)



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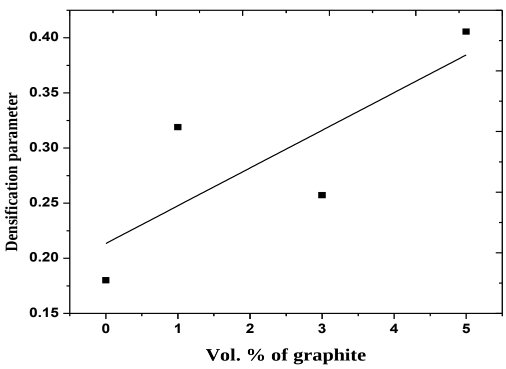


(c)

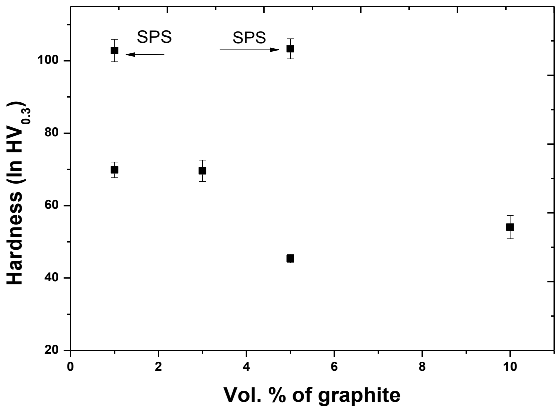
Fig.12 Variation of densification parameter at temperatures (a) 900 (b) 950 (c) 750°C after 1 h of sintering

4.3 Mechanical properties

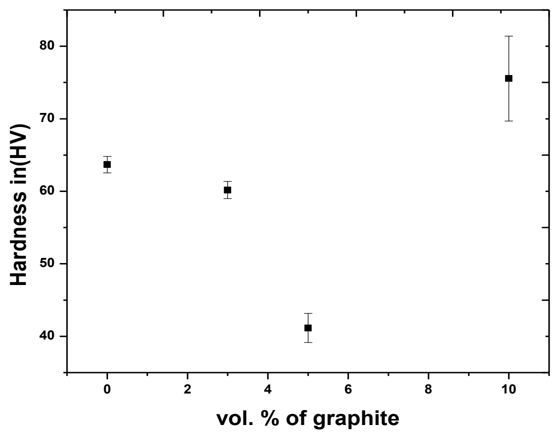
4.3.1 Hardness measurement

4.3.1.1 Effect of temperature

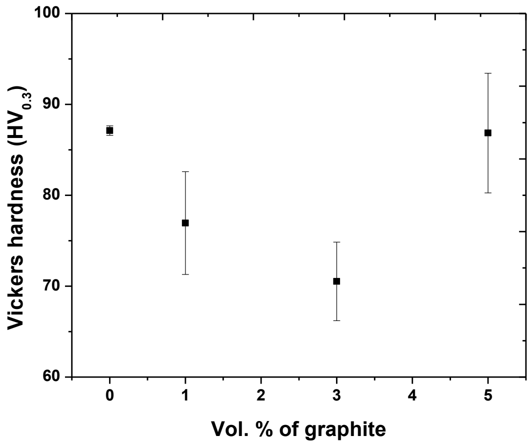
The variation in hardness with graphite reinforcements at different temperatures is shown in fig.13. The general hardness trend is that it decreases with increase in graphite percentage due to the soft nature of graphite. However, there are some hardness data which are uneven and scattered. The maximum Vickers hardness value of around 70 is achieved for 1vol. % graphite reinforced conventional sintered composite. Whereas higher hardness value of around 100 is obtained for spark plasma sintered composites due to simultaneous applications of heat and pressure during sintering. Another reason of higher hardness is fine grain size in case of SPS as it takes only 7 minutes for densification. The dispersion strengthening and grain size refinement are the main strengthening mechanism which is responsible for increase in hardness of Cu-graphite composites.



(a)



(b)

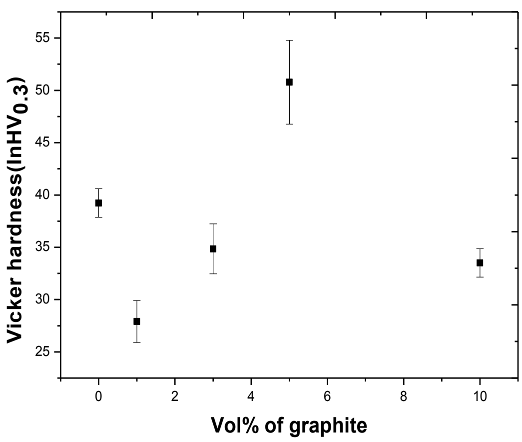


(c)

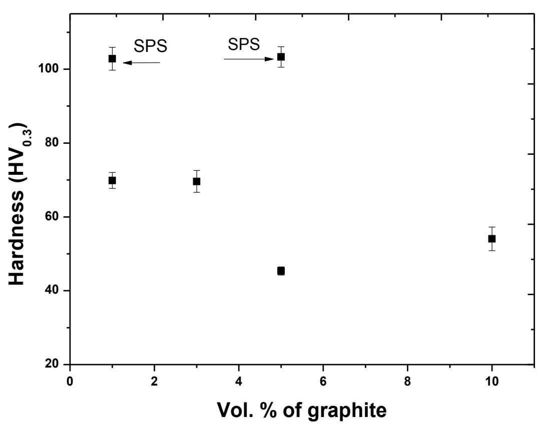
Fig.13 Variation in hardness with graphite percentage at temperatures of (a) 900(b) 950 & (c) 750°C

4.3.1.2 Effect of pressure

Fig. 14 shows the effect of compaction pressure on hardness of fabricated composites. From the graphs it is clear that maximum hardness is obtained at compaction pressure of 700 MPa which is in agreement with density value. So, it is the optimum pressure. But if pressure increases or decreases then it is seen that hardness decreases, which indicates that lesser hardening response of MMCs cold compacted at 600 and 800 MPa. The hardness values are also in agreement with density values at these two compaction pressures.



(a)



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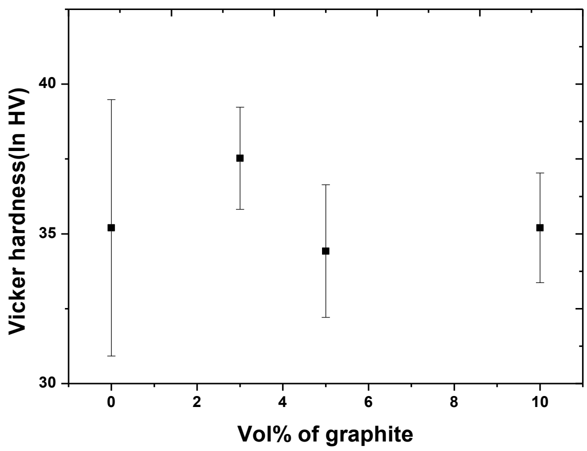
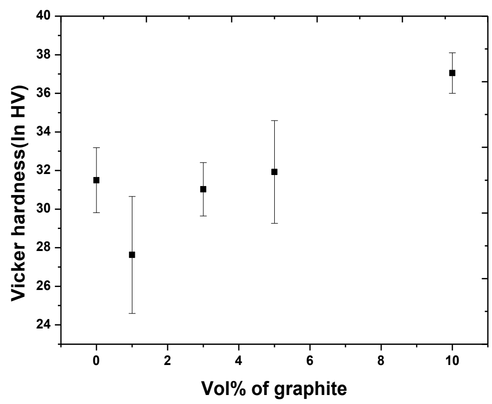


(c)

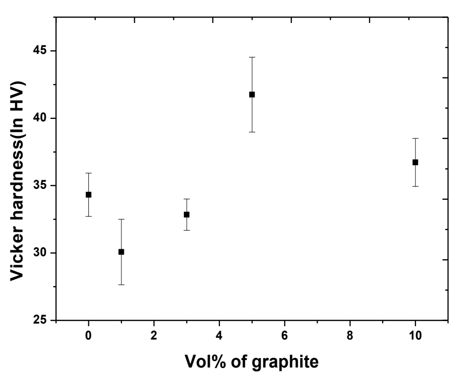
Fig. 14 Variation in hardness with graphite volume percentage at pressure (a) 600 (b) 700 (c) 800 MPa

4.3.1.3 Effect of time

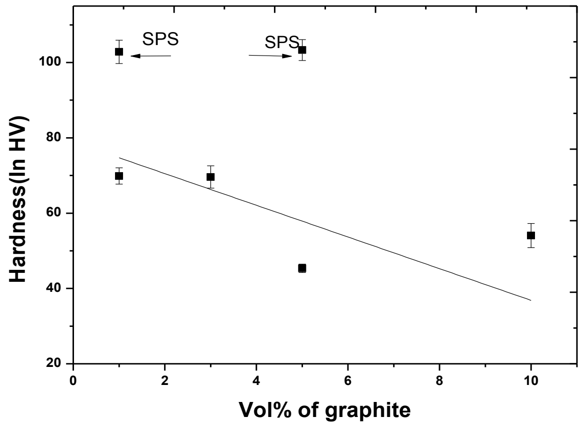
Fig.15 shows the hardness plots of Cu-graphite composites sintered at 900°C for 1.5, 1 and 0.5 h. It can be seen from hardness plots that hardness is maximum at 1 h of sintering time. So, 1 h is optimum sintering time. Sintering for 1.5 h results in coarsening of particles and sintering for 0.5 h results in under sintering. It also can be seen that maximum density is achieved for 1 h sintered sample and hence harness is maximum.



(a)



(b)

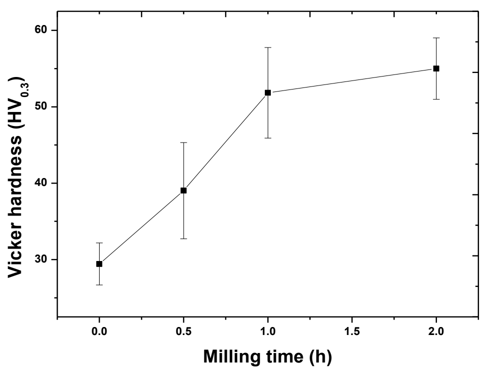


(c)

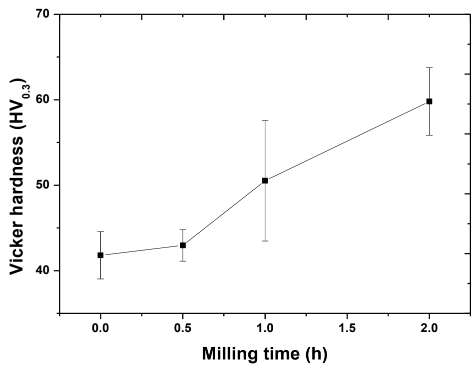
Fig. 15 Variation in hardness with graphite volume percentage at time (a) 1.5 (b) 0.5 (c) 1h

4.3.1.4 Effect of milling

Fig. 16 (a) & (b) show the effect of milling on Cu + graphite powder mixtures. It is found that with increase in milling time hardness of the composite increases. After milling the particle becomes fine and more closely bound and particle-particle contact increases with increase in fineness due to increase in surface area of the milled powder particle. Another reason of increasing hardness is that Cu powder particles are coated with graphite powder to some extent and improves the bonding between Cu and graphite.



(a)



(b)

Fig. 16 Variation of hardness for (a) Cu-1 vol. % graphite & (b) Cu-5 vol. % graphite composite with milling

4.3.2 Transverse rupture strength study

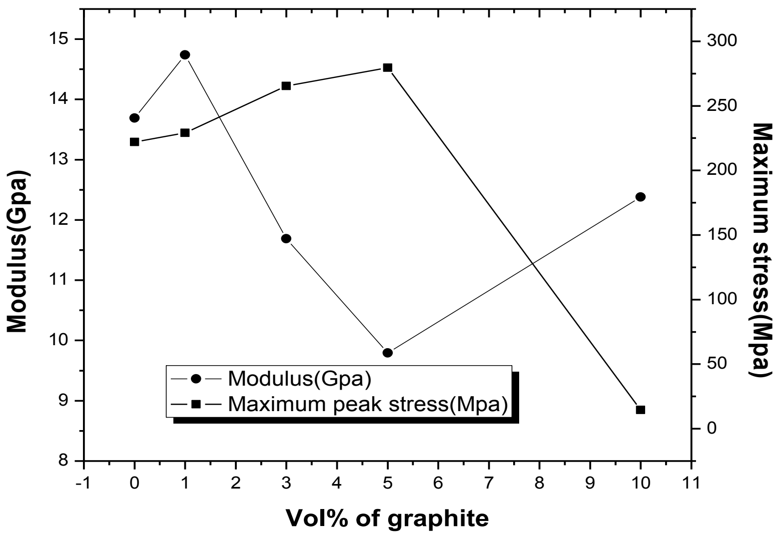
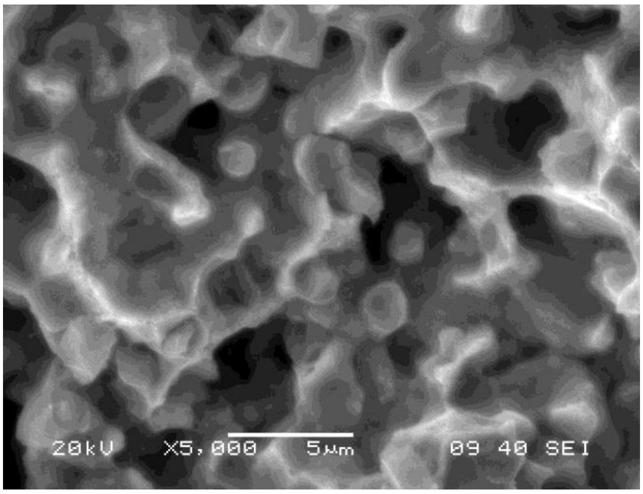
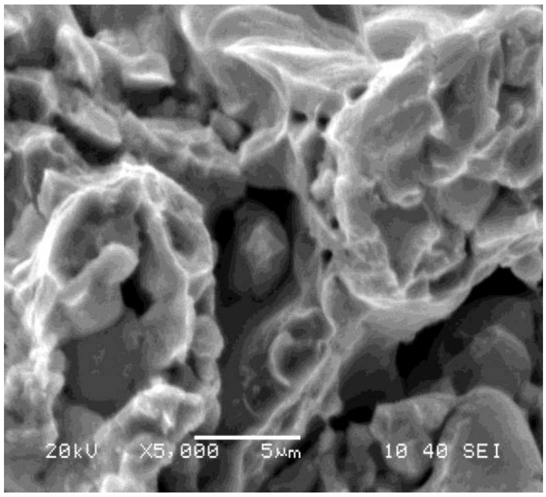


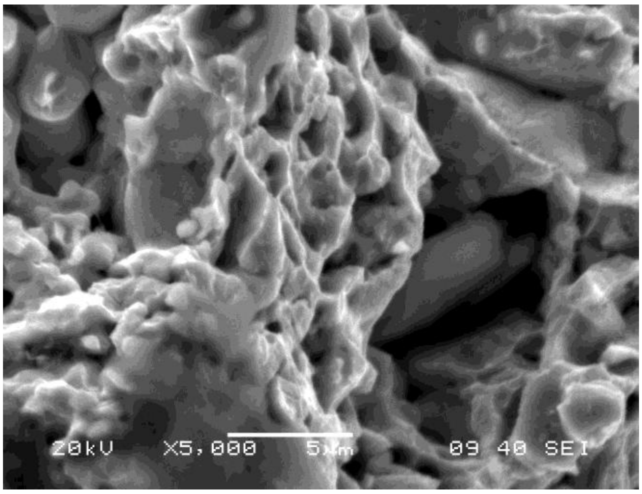
Fig. 17 Plot of flexural strength and elastic modulus with graphite content

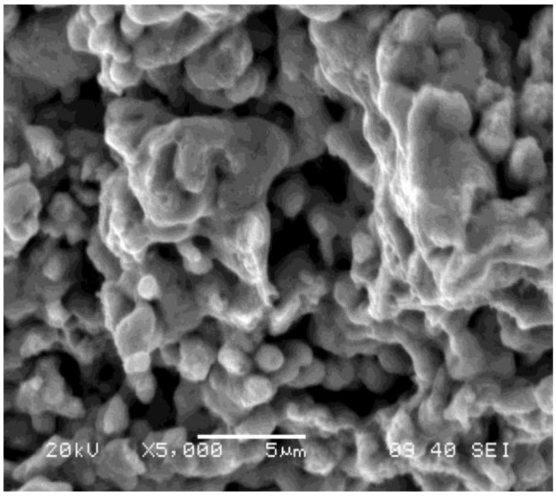
The flexural strength graph of Cu with 0, 1, 3, 5 and 10 vol. % of graphite composite fabricated by conventional sintering method for 1h are given in Figure 17. From this graph, it is concluded that minimum elastic modulus and maximum bending strengths are found in 5 vol. % of graphite reinforced composite. The maximum elastic modulus is achieved when the material is elastically deformed and its trend decreases at higher volume percentage of graphite. So it is concluded that, with higher volume percentage of graphite (> 5 vol. %) in composite is not preferable.





(a) (b)





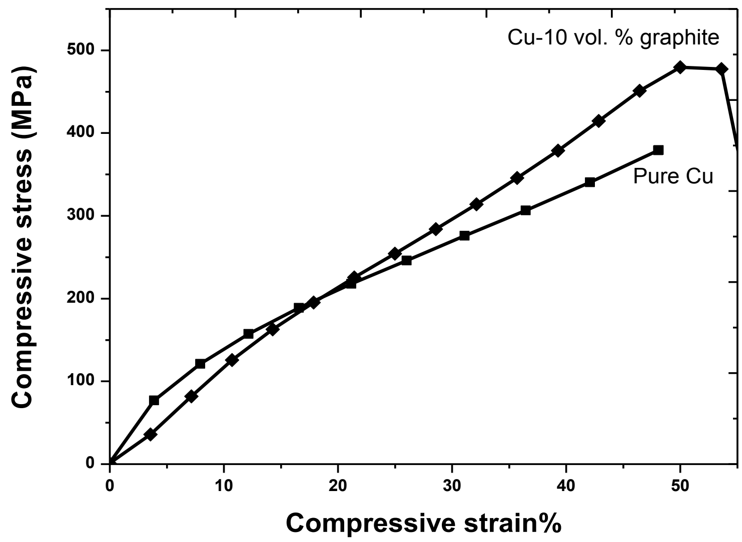
(c) (d)

Fig. 18 Micrographs of fracture surface of Cu with (a) 1 (b) 3 (c) 5 & (d)10 vol. % graphite reinforced MMC after TRS test

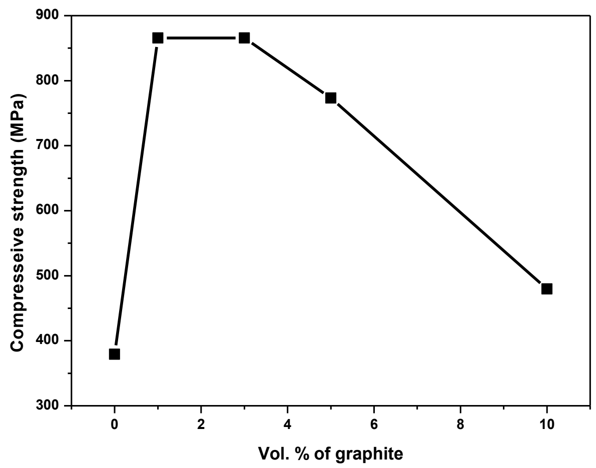
And from the micrographs of cu with 1, 3, 5 and 10 vol. % of TRS samples are shown in Figure 18(a-d). The present failure micro structural study shows the dimple size decreases with increases in graphite vol. % of graphite. It may be due to increase in brittleness with increase in percentage graphite.

4.3.3 Study of compressive strength

Compression strengths plots are shown in fig. 19 (a) & (b). It is found that maximum compression strength is found in 1 & 3 vol. % of graphite reinforced MMC. And it is suddenly decreases with further increase in graphite volume percentage due to brittle nature of composites. It also can be observed from the graph that at a particular amount of strain, compressive strength of Cu-10 vol. graphite composite is higher than pure Cu.



(a)



(b)

Fig. 19 Graphs for (a) compressive stress with strain & (b) compressive strength with volume percentage of graphite

The fracture surface of Cu-10 vol. % graphite reinforced composite tested under compression is shown in Fig. 20. It can be seen from the micrograph that de-bonding between the layers, crack initiation at voids/second phase particles/interface and propagation of crack results in failure of the composites under compression.

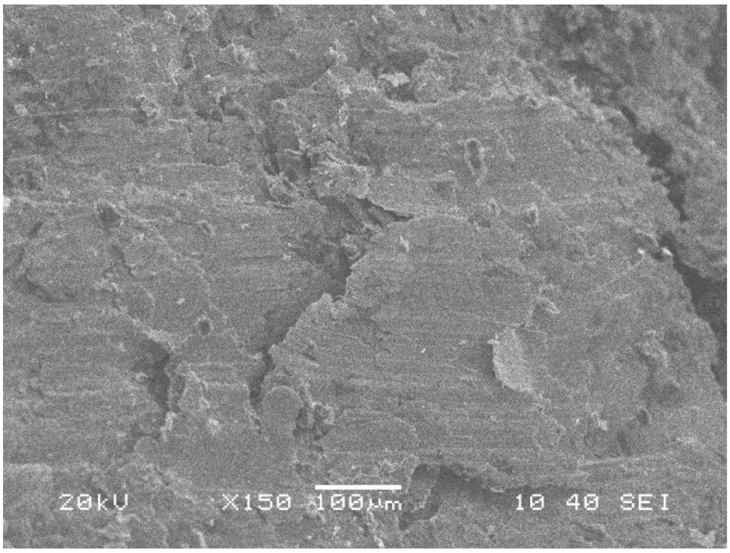


Fig. 20 SEM micrograph of compressive failure of Cu-10 vol. % graphite MMC

4.3.4 Wear study

From the micrographs (Fig. 21) we can observe that the wear resistance increases as per the following trend: *Pure Cu< Cu-5 vol. % graphite (CS) < Cu-1 vol. % graphite (SPS) <Cu-5 vol. % graphite (SPS)*

Graphite adheres to the wear surface, and a solid self-lubricating film comes into being on the wear surface. The contacts between metal and metal are transformed into the contacts between graphite film and metal or graphite film and graphite film. Therefore, the wear properties of Cu–graphite composites are greatly improved in comparison with those of pure copper.

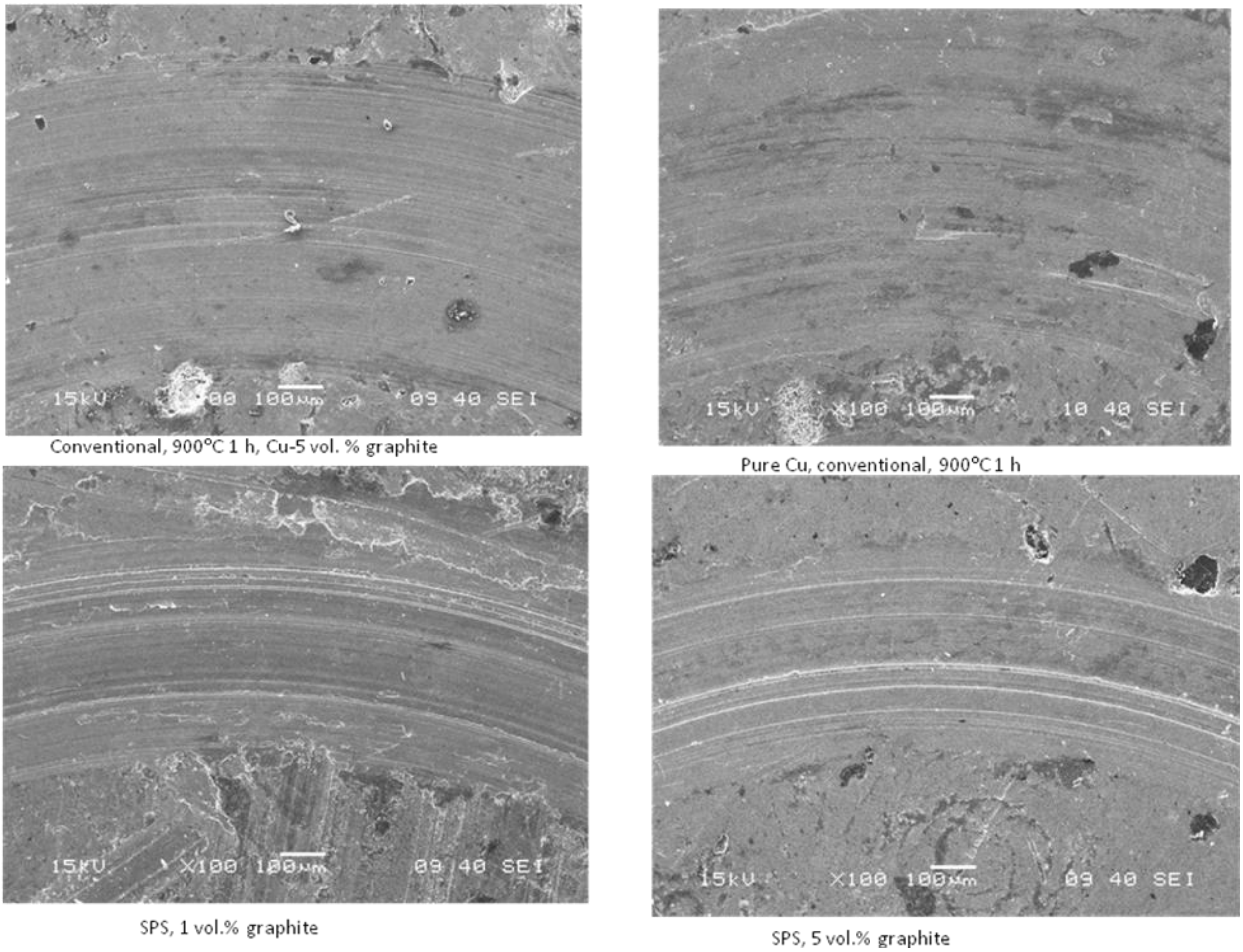
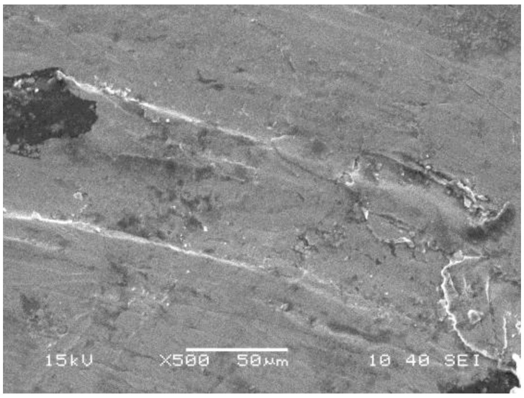
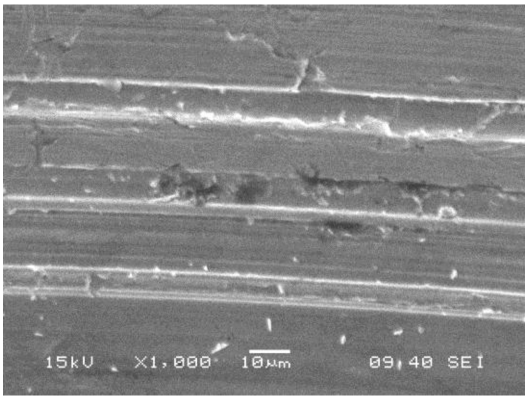


Fig. 21 Microstructure of rust surface of Cu with (a) conventionally sintered 5 vol. % of graphite,(b) pure copper,(c) SPS-1 & (d) SPS-5 vol. % graphite

Fig. 22 shows the enlarged view of worn surface of pure Cu and Cu-1 vol. % graphite reinforced composite. It is observed that some layer was delaminated from its surface (in case of conventionally sintered pure Cu), but in SPS sample the de-lamination is very less. Compatibility of SPS copper- graphite composite is better than conventionally sintered pure Cu due to presence of solid lubricant like graphite and evacuation in SPS processing.





(a) (b)

Fig. 22 Micrographs of failure surface of (a) pure copper & (b) SPS-1vol. % graphite.

Fig. 23 shows the wear depth of MMCs with time. An increase in trend of wear resistance and decrease in wear depth was observed for SPS sample with higher volume percent of graphite. The thickness of the messy graphite layer is the main key factor at the sliding surface of the wear specimen. That increases wear resistance of the composites.

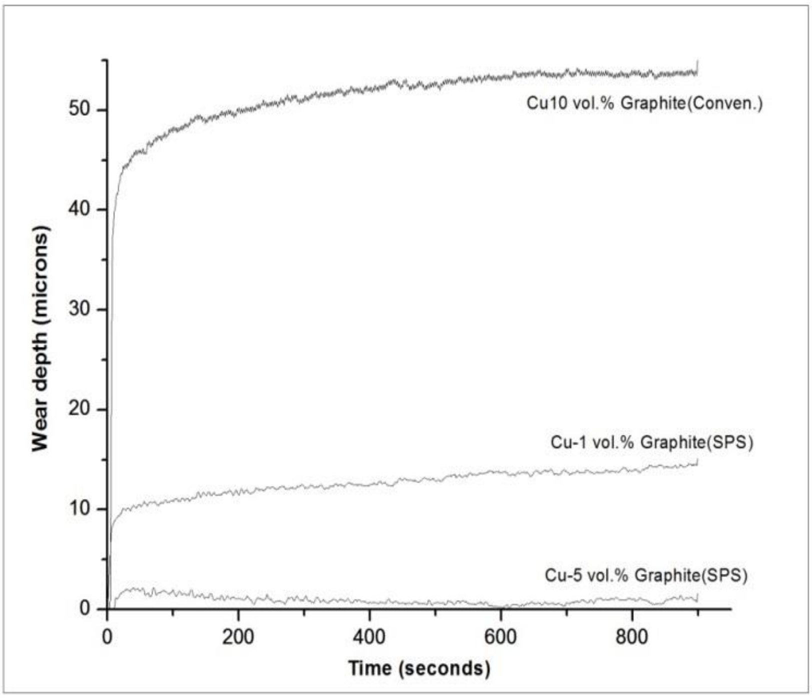


Fig. 23 Plot of wear depth with time for various composites

Chapter-5

Conclusion

Conclusions

The following conclusions can be drawn from the present investigation

- ❖ The copper-graphite composites were successfully fabricated by conventional as well as spark plasma sintering.
- ❖ Copper and carbon (graphite) with some copper oxide XRD peaks were seen in conventionally sintered composite, but in case of spark plasma sintered no oxide peaks were present due to evacuated condition.
- ❖ SEM analysis revealed the good bonding between copper matrix and graphite reinforcement.
- ❖ The optimum sintering temperature, pressure & time for conventional sintering are 900°C, 700 MPa, & 1h respectively.
- ❖ Milling of initial composite powder mixture results in very fine and homogeneously distribution of reinforcement throughout the matrix.
- ❖ The maximum transverse rupture strength and minimum modulus of elasticity is found at Cu with 5 vol. % of graphite reinforced MMC.
- ❖ The wear test shows that increase in thickness of messy graphite layer on the surface is the cause of decrease in wear track or depth.
- ❖ Maximum compressive strength is obtained at 1 & 3 vol. % of graphite reinforced MMC and then further addition of graphite leads to failure of the material due to increase in brittleness of the materials.
- ❖ Spark plasma sintered sample shows advanced properties than conventional sintered sample.

Chapter-6

Scope for future work

Scope for future work

- Pitch coke can be added to improve interfacial bonding between copper and graphite.
- Effect of load, coefficient of friction, wear rate, wear volume, wears mechanisms, etc. can also be studied.
- Electrical conductivity of Cu-graphite composites can also be measured.
- Cu-graphite MMC can also be fabricated by microwave sintering & hot pressing techniques and compared with the present study.

Chapter-7

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